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Sertifikaat   *Certificate*

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the documents annexed hereto are true copies of:

Application form P.1 , provisional specification and drawings of South African

Patent Application No. 2003/0484 as originally filed in the Republic of South

Africa on 17 January 2003 in the name of SASOL TECHNOLOGY

(PROPRIETARY) LIMITED for an invention entitled: " RECOVERY OF AN

ACTIVE CATALYST COMPONENT FROM A PROCESS STREAM ".

Geteken te ╻   in die Republiek van Suid-Afrika, hierdie   dag van
     PRETORIA   **22th**   **January 2004**
Signed at   in the Republic of South Africa, this   day of

....
Registrar of Patents

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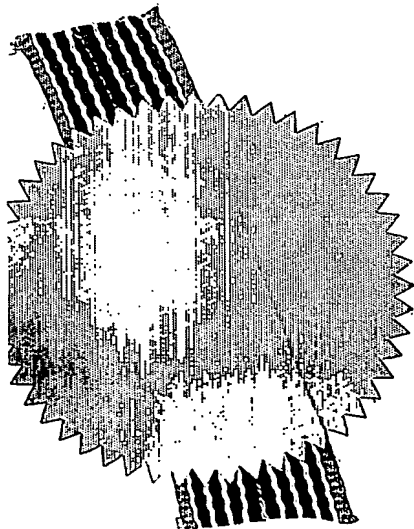
REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT AND
ACKNOWLEDGEMENT OF RECEIPT
(Section 30(1) Regulation 22)

FORM P.1
(to be lodged in duplicate)

THE GRANT OF A PATENT IS HEREBY REQUESTED BY THE UNDERMENTIONED APPLICANT
ON THE BASIS OF THE PRESENT APPLICATION FILED IN DUPLICATE

A&A REF   M45564 GSK

| 21 | 01 | PATENT APPLICATION 2003/0484 |

| 71 | FULL NAME(S) OF APPLICANT(S) |

SASOL TECHNOLOGY (PROPRIETARY) LIMITED

| ADDRESS(ES) OF APPLICANT(S) |

1 Sturdee Avenue, Rosebank, Johannesburg, Republic of South Africa

| 54 | TITLE OF INVENTION |

"RECOVERY OF AN ACTIVE CATALYST COMPONENT FROM A PROCESS STREAM"

Only the items marked with an "X" in the blocks below are applicable.

[ ] THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. The earliest priority claimed is

Country: No: Date:

[ ] THE APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO | 21 | 01 |

[ ] THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON

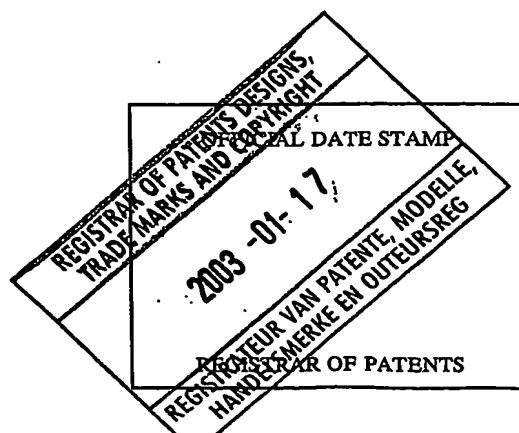APPLICATION NO | 21 | 01 |

THIS APPLICATION IS ACCOMPANIED BY:

X	A single copy of a provisional specification of **16** pages
X	Drawings of **3** sheets
	Publication particulars and abstract (Form P.8 in duplicate) (for complete only)
	A copy of Figure of the drawings (if any) for the abstract (for complete only)
	An assignment of invention
	Certified priority document(s). (State quantity)
	Translation of the priority document(s)
	An assignment of priority rights
	A copy of Form P.2 and the specification of RSA Patent Application No	21	01	
X	Form P.2 in duplicate
	A declaration and power of attorney on Form P.3
	Request for ante-dating on Form P.4
	Request for classification on Form P.9
	Request for delay of acceptance on Form P.4
	Extra copy of informal drawings (for complete only)

| 74 | ADDRESS FOR SERVICE: Adams & Adams, Pretoria |

Dated this **17** day of **January 2003**

ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS

The duplicate will be returned to the applicant's address for service as
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A&A P201

OFFICIAL DATE STAMP

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REPUBLIC OF SOUTH AFRICA
Patents Act, 1978

PROVISIONAL SPECIFICATION
(Section 30 (1) - Regulation 27)

21	01	OFFICIAL APPLICATION NO

22	LODGING DATE

ß2003/0484

17 January 2003

71	FULL NAME(S) OF APPLICANT(S)

SASOL TECHNOLOGY (PROPRIETARY) LIMITED

72	FULL NAME(S) OF INVENTOR(S)

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54	TITLE OF INVENTION

"RECOVERY OF AN ACTIVE CATALYST COMPONENT FROM A PROCESS STREAM"

5

THIS INVENTION relates to the recovery of an active catalyst component from a process stream. More particularly, the invention relates to a

10 process for recovering an active catalyst component from a process stream, and to a hydroformylation process.

According to a first aspect of the invention, there is provided a process for recovering an active catalyst component from a process stream

15 containing, in addition to the active catalyst component, also at least aldols, acetals and/or esters, which process includes

admixing at least one C_1 to C_{10} alcohol with the process stream;

allowing an active catalyst component to precipitate; and

separating the precipitated active catalyst component from a

20 residual alcohol-rich phase.

The process stream may, in particular, be a hydroformylation process heavies purge stream, as hereinafter described.

25 The hydroformylation process (or Oxo process) is a catalytic process for the conversion, at elevated temperature and pressure, of an olefin into an aldehyde and/or alcohol product having one carbon more than the starting olefin by the addition of one molecule each of hydrogen and carbon monoxide to the carbon-carbon double bond of the olefin, thereby adding

30 a carbonyl or carbinol group to an olefinically unsaturated carbon atom and saturating the olefin bond.

The hydroformylation catalyst is selected according to the particular oxygenated products which are required from a particular olefinic feedstock.

5   The catalyst used is, however, typically a homogeneous (liquid phase) catalyst, and may comprise a mixture or complex of a metal, for example, cobalt (Co), rhodium (Rd), ruthenium (Ru) or palladium (Pd); carbon monoxide; and a phosphine and/or phosphate ligand. Examples of such catalysts are triphenyl phosphine ligands used with rhodium, and alkyl

10   phosphine ligands used with cobalt. Specific examples of the latter are trialkyl phosphines and bicyclic tertiary phosphines such as 9-phosphabicyclo [3.3.1] nonane and 9-phosphabicyclo [4.2.1] nonane represented by formulas (I) and (II) respectively:

15

P—$C_{20}H_{41}$

P—$C_{20}H_{41}$
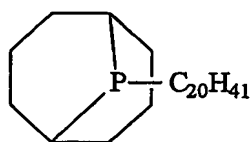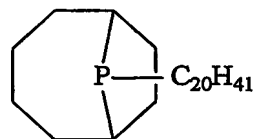
I   **II**

20

The ligands I and II are available commercially, as a mixture, under the collective chemical name eicosyl phoban ('EP').

The catalyst mixture or complex is preferably used as an admixture with

25   an aromatic, olefinic and/or paraffinic organic solvent. The organic solvent may thus be a common solvent such-as toluene, or a long chain paraffin. However, the olefinic feedstock of the hydroformylation process in which the catalyst is to be used, can also be used as the organic solvent, if desired.

30

Thus, in the hydroformylation process, an olefin-containing feedstock is reacted, in a reaction zone and in the presence of the catalyst, and at

elevated temperature and pressure, with carbon monoxide and hydrogen, with olefins in the feedstock thus reacting with carbon monoxide and hydrogen to form aldehydes and alcohols; a reaction mixture comprising unreacted feedstock, alcohols, aldehydes, catalyst residue and, optionally,

5   unreacted gaseous reactants, is withdrawn from the reaction zone and passed to a separation zone to separate liquid and gaseous phases; the liquid phase passes to a distillation zone where the alcohols, aldehydes and unreacted feedstock are separated from the catalyst residue; the catalyst residue is withdrawn as a bottoms component and recycled to

10   the reaction zone; and the alcohols, aldehydes and unreacted feedstock are withdrawn from the distillation zone as an overheads component.

The catalyst residue thus contains the active catalyst component, which may be the metal/carbon monoxide/ligand complex, and, optionally, free

15   ligand.

During the hydroformylation reaction, heavies or bottoms high boiling by-products are formed as a result of reactions between the products. Specifically, aldols, acetals and esters are formed during the

20   hydroformylation reaction. More specifically, aldols are formed as a result of -aldol condensation between two aldehyde molecules. Esters are formed as a result of the reaction between acids (resulting from the metal in the catalyst) and alcohols. Finally, acetals are formed as a result of the reaction between alcohols and aldehyde molecules formed during the

25   reaction.

Since these products are not removed in the distillation zone overheads component, they are recycled back to the reaction zone as part of the bottoms component. With continuous recycle, there is thus a continuous

30   build-up of the heavies in the reactor. Over time these heavies will build up to unacceptable levels if not removed. Typically, a constant level of 25% (mass basis) heavies content in the reaction zone is required. In

order to keep the heavies level constant, they need to be removed at the same rate as that in which they are formed. This is accomplished by removing (purging) part of the bottoms component or recycle stream, eg by means of a pump, as the heavies purge stream hereinbefore referred
5   to.

The continuous purging of heavies also leads to a loss of expensive catalyst, ie metal/carbon monoxide/ligand complex, and, optionally, free ligand, from the hydroformylation cycle. Although the ligand and catalyst
10 lost during purging can be replenished, the continuous loss and lack of recovery of the catalyst and ligand can have severe implication to the process economics, especially if an expensive ligand and/or catalyst source is used. Thus, such loss, on a commercial scale operation, represents a stiff economic penalty which can make the process
15 prohibitively expensive. It is for this reason that a cost effective quantitative method to recover the catalyst and/or optionally free ligand from the heavies is highly desirable in the art. The present invention provides such a cost-effective quantitative method.

20 In addition, the loss of catalyst and ligand has severe environmental implication in considering the poisonous nature of the catalyst metal (eg cobalt) and phosphorus based ligand.

An added advantage of the process of the invention is that the inactive
25 catalyst component, namely ligand phosphine oxide, remains in the residual alcohol-rich phase, thus circumventing the build-up of the oxide in the reaction zone over time. The heavies also remain in the residual alcohol-rich phase. The heavies can be recovered from the residual alcohol-rich phase, and the recovered heavies could then, in the absence
30 of catalyst and free ligand, be cracked back to the product components (alcohols and aldehydes), adding another economic benefit to the process.

According to a second aspect of the invention, there is provided a hydroformylation process, which includes

reacting, in a reaction zone and in the presence of a
5   hydroformylation catalyst, and at elevated temperature and pressure, an olefin-containing feedstock with carbon monoxide and hydrogen, to form aldehydes and alcohols;

withdrawing a reaction mixture comprising the alcohols, the aldehydes, unreacted feedstock, catalyst residue, heavies and, optionally,
10   unreacted gaseous reactants, from the reaction zone;

in a separation zone, separating a gaseous phase from a liquid phase comprising the aldehydes, alcohols, unreacted feedstock, the heavies and the catalyst residue;

in a distillation zone, subjecting the liquid phase to distillation;
15   withdrawing from the distillation zone, as an overheads component, the alcohols, aldehydes and unreacted feedstock;

withdrawing from the distillation zone, as a bottoms component, the heavies and the catalyst residue;

admixing at least one C_1 to C_{10} alcohol with at least a portion of the
20   bottoms component;

allowing an active catalyst component to precipitate; and

separating the precipitated active catalyst component from a residual alcohol-rich phase.

25   The hydroformylation catalyst may be as hereinbefore described.

The process may include recycling a portion of the bottoms component to the reaction zone, with the portion thereof that is admixed with the C_1 to C_{10} alcohol thus constituting a heavies purge stream that is withdrawn.

30

The process of the invention is characterized thereby that no dilution of the bottoms component, at least prior to the withdrawal of the heavies

purge stream therefrom, or of the heavies purge stream, with a saturated or unsaturated aliphatic hydrocarbon having 3 to 20 carbon atoms or with an aromatic or hydrocarbyl-substituted aromatic hydrocarbon having from 6 to 22 carbon atoms, takes place.

5

Thus, the addition of the C_1 to C_{10} alcohol to the heavies purge stream leads to the selective precipitation of the metal/carbon monoxide/ligand complex and, optionally, free phosphorus ligand. As mentioned hereinbefore, the catalyst residue will normally contain, in addition to the

10 active catalyst component, also inactive catalyst components such as phosphine ligand oxides. These phosphine ligand oxides and heavies will not precipitate out, but will remain in the alcohol-rich phase.

The C_1 to C_{10} alcohol may, in particular, be a monohydric alcohol or a

15 dihydric alcohol, ie an alcohol containing two hydroxyl groups.

When a monohydric alcohol is used, it preferably has a C_1 to C_8 carbon chain; more preferably, it is then a concentrated or undiluted primary alcohol having 1 to 3 carbon atoms. Most preferably, concentrated or

20 undiluted methanol or ethanol is then used.

When a dihydric alcohol is used, it preferably has a C_2 to C_8 carbon chain. For example, undiluted or concentrated ethylene glycol or propylene glycol can then be used.

25

The Applicant has also found that while a single monohydric or dihydric alcohol can be used for efficient ligand and/or catalyst recovery, even more efficient recoveries can, in certain circumstances, be achieved by using a mixture of two or more undiluted or concentrated alcohols. For

30 example, an undiluted dihydric alcohol such as ethylene glycol or propylene glycol can effectively be used in admixture with at least one other dihydric alcohol and/or with at least one monohydric alcohol. The

alcohol mixture will then naturally be admixed with the process stream or with the bottoms component, as the case may be.

It has thus been discovered that the addition of a C_1 to C_{10} alcohol, and
5   more specifically a C_1 to C_5 alcohol, such as a C_1 to C_3 monohydric alcohol and/or a C_2/C_3 dihydric alcohol, to the heavies purge stream leads to the recovery of an active catalyst component, viz metal/carbon monoxide/ligand complex, and, optionally, free ligand.

10   Although the alcohol can be at room temperature, it has unexpectedly been found that the use of chilled alcohol, and in particular chilled methanol, results in particularly effective precipitation of the active catalyst component. The alcohol is thus preferably at a temperature below room temperature, more preferably at a temperature below $0°C$, eg
15   at about $-5°C$. Naturally, the alcohol must be in liquid form, and it will thus be at a temperature above its freezing temperature.

The process may include recycling the precipitated active catalyst component to the reaction zone.
20

The alcohol-rich phase can also be processed further, if desired, eg to recover the alcohol for re-use.

The reaction temperature may be from $100°C$ to $300°C$, typically from
25   $150°C$ to $200°C$.

The reaction pressure may be at least 20 bar (150psi), preferably between 50 bar (750psi) and 100 bar (1500psi), typically about 85 bar (1232psi).

30

The hydroformylation reaction zone may be provided by a reactor capable of handling a homogenously catalysed chemical transformation, such as a continuous stirred tank reactor ('CSTR'), bubble column, or the like.

5   The olefin-containing feedstock may, in particular, be a C_2 to C_{20} Fischer-Tropsch derived olefin stream. Thus, the olefin-containing feedstock may be that obtained by subjecting a synthesis gas comprising carbon monoxide and hydrogen to Fischer-Tropsch reaction conditions in the presence of an iron-based, a cobalt-based or an iron/cobalt-based Fischer-
10 Tropsch catalyst, with the resultant olefinic product then constituting the olefin-containing feedstock of the process of the invention, or a component thereof constituting the olefin-containing feedstock of the process of the invention.

15 In other words, the olefinic product from the Fischer-Tropsch reaction can, if necessary, be worked up to remove unwanted components therefrom and/or to separate a particular olefinic component therefrom, with said particular olefinic component then constituting the olefin-containing feedstock of the process of the invention.
20

The invention will now be described in more detail, with reference to the accompanying drawings.

In the drawings,
25   FIGURE 1 shows a flow diagram of a hydroformylation process demonstration unit ('PDU');

FIGURE 2 shows a ^{31}P NMR spectrum of the purge derived precipitate obtained in Example 1;

FIGURE 3 shows an IR spectrum of the purge derived precipitate
30 obtained in Example 1.

In the drawings, reference numeral 10 generally indicates a flow diagram of a hydroformylation or oxo process demonstration unit ('PDU'). The PDU is used to test the continuous operation of the oxo process.

5   The PDU 10 includes an olefin-containing feedstock container 12 connected, by means of a feed line 14, to a feed pot 16. A flow line 18, fitted with a feed pump 20, leads from the feed pot 16 to a hydroformylation reactor 22.

10   The reactor 22 is fitted with a stirrer 24, and a syngas feed line 26 also leads into the reactor 22. A reaction mixture withdrawal line 28 leads from the reactor 22 to a knockout pot 30, with a flow line 32 leading from the knockout pot 30 to a level pot 34. A gas phase withdrawal line 36 leads from the level pot 34 as does a liquid phase withdrawal line 38.

15   The line 38 leads to a feed pot 40, with a flow line 42, fitted with a feed pump 44, leading from the feed pot 40 into a short path distillation ('SPD') unit 46. An overheads line 48, for withdrawing an overheads component, leads from the unit 46 as does a bottoms component withdrawal line 50. The line 50 is fitted with a discharge pump 52 and

20   leads to a purge pot 54.

The PDU 10 includes a catalyst feed pot 56, with a flow line 58 leading from the feed pot 56 to the purge pot 54. A catalyst recycle line 60, fitted with a recycle pump 62, leads from the purge pot 54 back to the

25   reactor 22.

In use, a Fischer-Tropsch derived olefin-containing feedstock containing a range of alpha alkenes, is fed to the reactor 22 from the feed pot 16. Syngas, containing carbon monoxide and hydrogen, is also fed into the

30   reactor 22 by means of the flow line 26. The reactor 22 contains a homogeneous (liquid phase) catalyst comprising a mixture or complex of

cobalt, carbon monoxide and phosphine ligand (EP) mixed with toluene as an aromatic organic solvent.

The alkenes react with the carbon monoxide and hydrogen to form
5   aldehydes and alcohols, in accordance with the so-called oxo reaction. The hydroformylation reaction typically takes place at 85 bar and 170°C.

A reaction mixture comprising aldehydes, alcohols, unreacted feedstock, heavies, catalyst residue and unreacted gaseous reactants, leaves the
10   reactor 22 along the flow line 23 and passes into the knockout pot 30 and then into the level pot 34 where liquid and gas phases are separated. The gas phase is withdrawn along the flow line 36, while the liquid phase, comprising unreacted feedstock, aldehydes, alcohols, heavies and catalyst residue, is withdrawn along the flow line 38. The liquid phase is
15   depressurized across a level control valve in the level pot 34 and then passes to the feed pot 40. From the feed pot 40, the liquid phase is pumped, by means of the line 42 and pump 44, to the SPD unit 46, where the alcohols, aldehydes and unreacted olefin-containing feedstock are separated from the heavies and the catalyst residue, and withdrawn
20   as an overheads component, along the flow line 48. The heavies and catalyst residue are withdrawn, as a bottoms component, and pass along the flow line 50 to the purge pot 54, where makeup catalyst is added thereto from the catalyst feed pot 56. The bottoms component then passes, from the purge pot 54, back to the reactor 22, by means of the
25   line 60 and the pump 62. The catalyst residue comprises active catalyst components, namely cobalt/carbon monoxide/ligand complex and, optionally, ligand on its own, together with inactive catalyst components such as ligand oxide.

30   The PDU 10 is thus a mini pilot plant or rig which is used to test a hydroformylation process on a large scale, before upgrading to pilot plant scale.

During the hydroformylation reaction, heavies (high boiling by-products) are formed as hereinbefore described. These heavies are not removed in the SPD unit 46, and are thus recycled back to the reactor 22 together

5 with the catalyst residue. Over time, these heavies build up to unacceptable levels, if not removed. Typically, a constant level of 25% (mass basis) heavies content in the reactor 22 is required. In order to keep the heavies level in the reactor 22 constant, they need to be removed at the same rate at which they are formed. This is accomplished

10 by removing (purging) part of the recycle stream, along a purge line 64 fitted with a pump 66. The line 64 leads into a container 68 with a nitrogen line 70 also leading into the container 68. The purge stream is thus collected in the container 68 that is blanketed with nitrogen to prevent the purge stream from oxidizing. The purge stream can be

15 recovered from the container 68 for further investigation.

The continuous purging of heavies also leads to a loss of expensive catalyst complex and/or ligand from the hydroformylation process, as hereinbefore described. Thus, although the ligand and catalyst lost during

20 the purging can be replenished, the continuous loss and lack of recovery thereof can have severe implication on process economics. For example, an 8 kilo ton/annum purge stream from a commercial hydroformylation plant would contain 422 ton/annum of phosphine which could be lost if not recovered efficiently.

25

The catalyst addition system comprising the catalyst feed pot 56 and the line 58 was installed, in the PDU unit 10, primarily to replace catalyst that is purged via the purge line 64. However, the catalyst addition system, since it is a closed system, also prevents any oxidation of the catalyst.

30 The catalyst addition system can also be used at start-up to introduce catalyst into the process.

The purge stream withdrawn along the line 64 is thus a mixture of heavies, active cobalt/carbon monoxide/ligand complex or catalyst, and, optionally, free phosphine ligand and inactive phosphine ligand oxide.

5   This purge stream was treated with alcohol in accordance with the invention, as described in Example 1 hereunder.

EXAMPLE 1

Typically, 50mℓ of the purge stream from the PDU unit 10 was treated,
10   in a 500mℓ separating funnel, with 250mℓ to 300mℓ concentrated or undiluted methanol (100%) chilled to about -5°C. After vigorous shaking, the solution was allowed to separate out over a period of 10 to 30 minutes.   During this period, a golden yellow precipitate component settled out of the bottom of the flask. This precipitate component was
15   separated from the residual methanol phase and subjected to careful filtration under argon, to recover an active catalyst component, namely cobalt/carbon monoxide/phosphine ligand complex. The active catalyst component was dried at room temperature under a flow of argon gas. The recovered catalyst component was then weighed and analyzed by ^{31}P
20   NMR and infrared ('IR') spectroscopy. The methanol phase was evaporated off to recover the methanol, leaving a residue containing heavies and phosphine ligand oxide.

Figure 2 is the ^{31}P NMR spectrum for the recovered catalyst precipitate
25   after drying, while Figure 3 is an infrared (IR) spectrum of the recovered precipitate. In Figure 2, the peak at 1.88 and -38pm is for free phosphine ligand (EP). In Figure 3, the peak assignments are as follows: 1921,1950cm^{-1}: $Co_2(CO)_6EP_2$, 1968cm^{-1}: $HCo(CO)_3EP$, 2028cm^{-1}: $Co_2(CO)_7EP$.

30

Re-use of the recovered catalyst precipitate, ie the recovered cobalt/carbon monoxide/phosphine ligand complex, was demonstrated in

Examples 2 to 4. In Examples 2 to 4, the reactor that was used in each case was a Parr autoclave reactor, ie a batch reactor.

EXAMPLE 2

5   In a typical experiment, 1.5g of the catalyst precipitate of Example 1 was introduced into the 100mℓ autoclave to yield a cobalt concentration of 1000ppm. 50mℓ of the olefin feedstock, namely depenternizer overheads (DPO), was introduced. DPO is a Fischer-Tropsch derived feed which contain C_5 olefins including 70% 1-pentene. The reaction mixture

10   was then heated to 170°C. After this temperature was reached, the mixture was pressurized to 75 bar with syngas having a 2:1 H_2:CO molar ratio. The reaction mixture was then left to react for 3 hours with syngas fed on demand. After the allotted time, the mixture was cooled to room temperature, and thereafter a sample of the reaction mixture was

15   obtained and analyzed by GC and GC-MS.

Results:

From Catalyst re-use: % 1-pentene conversion: 97.3%, C_6 alcohol linearity = 85.6%: n:i = 15.8.

From PDU run (Example 1): % 1-pentene conversion: 98.2%, C_6 alcohol

20   linearity = 87.9%: n:i = 20.1.

EXAMPLE 3

In a typical experiment, 1.5g of the catalyst precipitate of Example 1 was introduced into the 100mℓ autoclave. 50mℓ of olefin feedstock, namely

25   100% 1-pentene, was introduced into the autoclave. The solution was then heated to 170°C. After this temperature was reached, the mixture was pressurized to 75 bar with syngas having a 2:1 H_2:CO molar ratio. The reaction mixture was then left to react for 3 hours with syngas fed on demand. After the allotted time, the mixture was cooled, and

30   thereafter reacted, sampled and analyzed by GC and GC-MS.

Results:

From Catalyst re-use (precipitate): % 1-pentene conversion: 97.1%, C_6 alcohol linearity = 93.4%: n:i = 17.6.

*From freshly prepared catalyst run: % 1-pentene conversion: 95.4%, C_6 alcohol linearity = 95.4%: n:i = 19.8.

5

* Cobalt Concentration = 1000ppm. Ligand:Metal ratio = 3:1. This run was then also conducted in the Barr autoclave; all conditions were identical to those using the recovered catalyst precipitate except that fresh catalyst was used.

10

EXAMPLE 4

In a typical experiment, 1.5g of the catalyst precipitate of Example 1 was introduced into the 100ml autoclave. 50ml of olefin feedstock, namely C_7 olefin, 1-heptene, was introduced into the autoclave. The solution was

15   then heated to 170°C. After this temperature was reached, the mixture was pressurized to 75 bar with syngas having a 2:1 H_2:CO molar ratio. The reaction mixture was then left to react for 3 hours with syngas fed on demand. After the allotted time, the mixture was cooled, and thereafter reacted, sampled and analyzed by GC and GC-MS.

20   Results:

From Catalyst re-use: % 1-heptene conversion: 98.4%, C_8 alcohol linearity = 87.9%: n:i = 18.5.

*From freshly prepared catalyst run: % 1-heptene conversion: 97.5%, C_8 alcohol linearity = 86.6%: n:i = 18.6

25

*Cobalt Concentration = 1000ppm. Ligand:Metal ratio = 3:1. This run was then also conducted in the Barr autoclave; all conditions were identical to those using the recovered catalyst precipitate except that fresh catalyst was used.

30

It has thus been shown that a catalyst precipitate, ie active catalyst components, recovered in accordance with the invention, can be re-used

in batch autoclave runs. It has also been shown that the recovered catalyst precipitate can be used in a continuous demonstration unit process without any marked or significant decrease in catalyst activity and/or product quality and/or selectivity.

5

DATED THIS 17TH DAY OF JANUARY 2003.
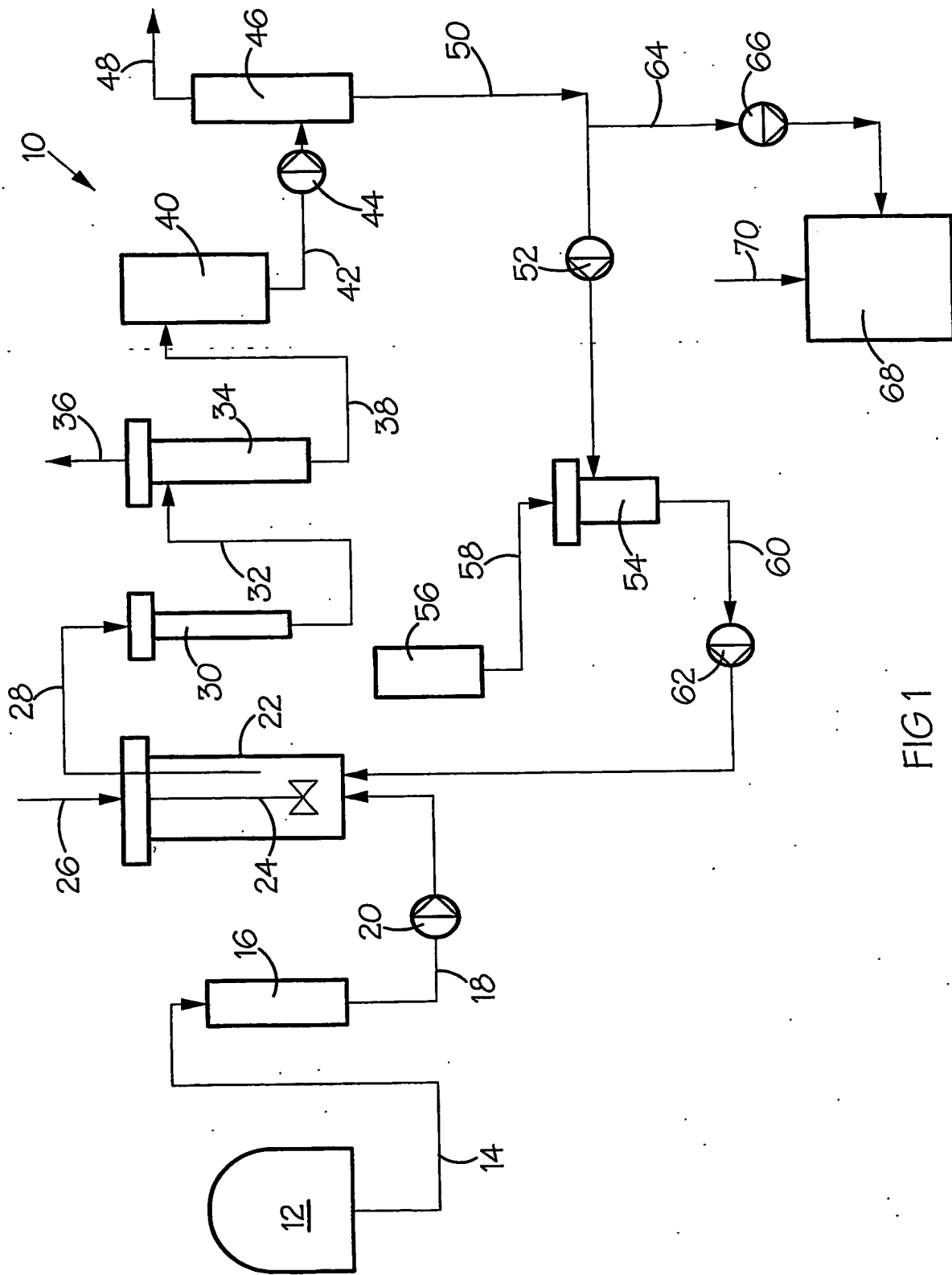
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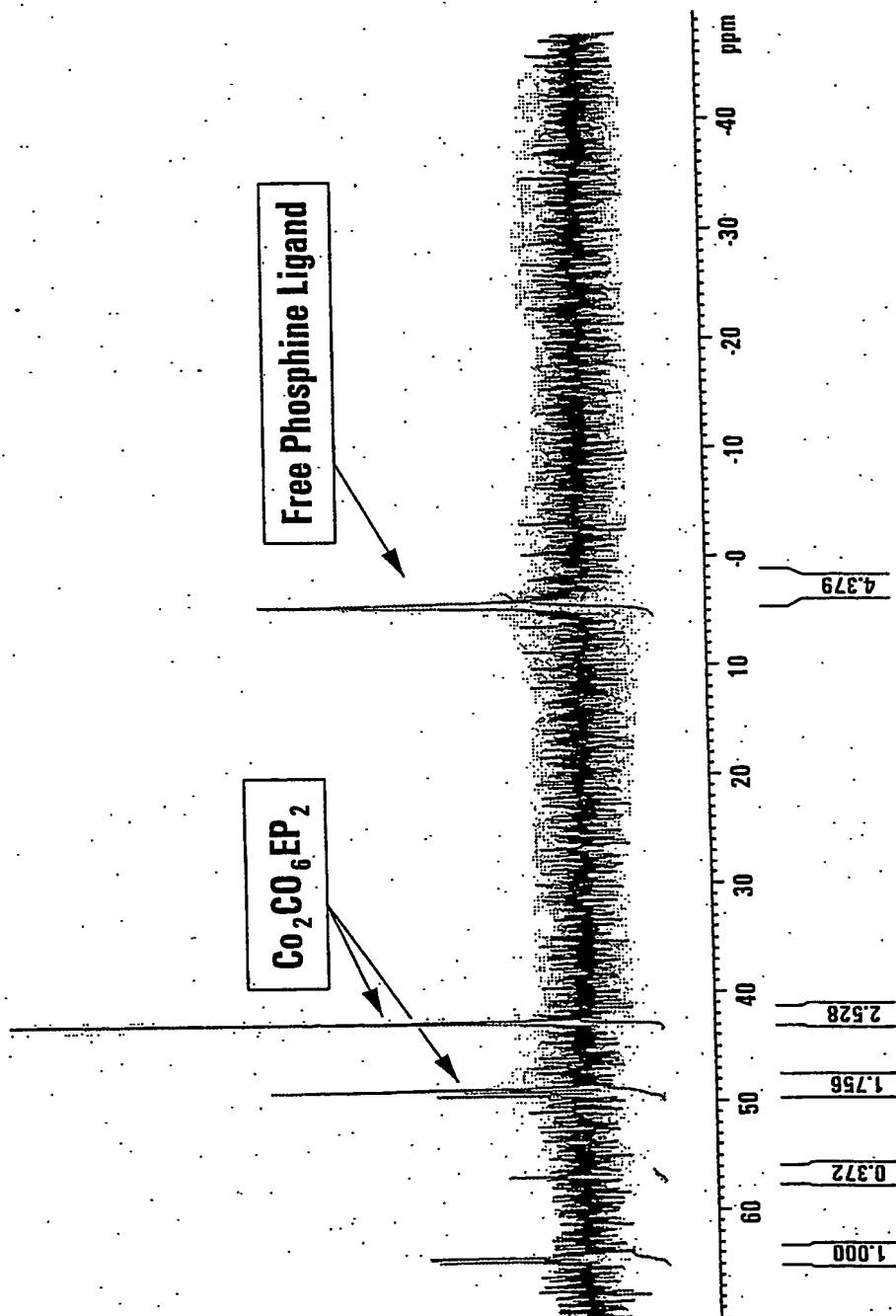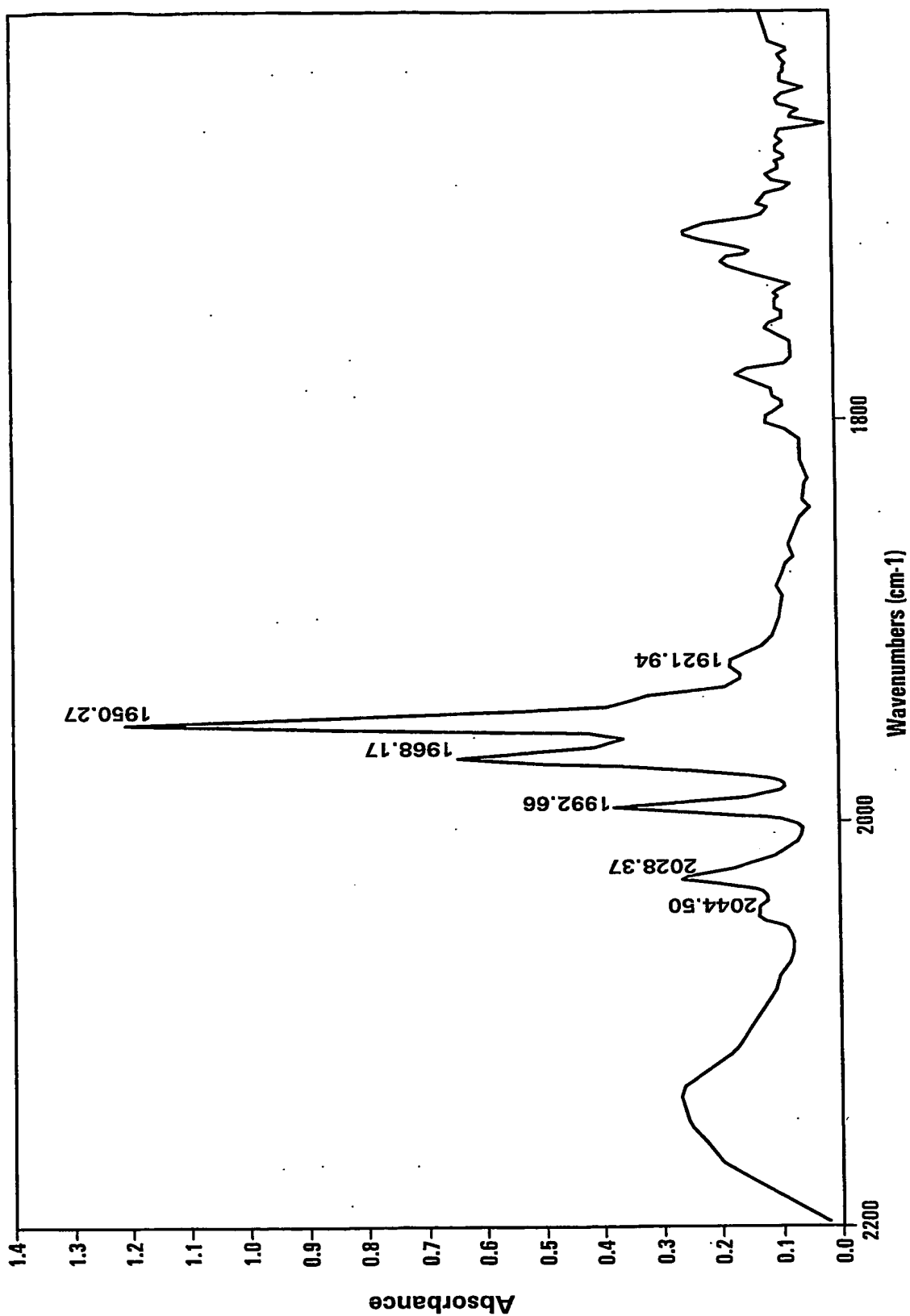APPLICANTS PATENT ATTORNEYS

10

SASOL TECHNOLOGY (PROPRIETARY) LIMITED

FIG 1

ADAMS & ADAMS

Free Phosphine Ligand

$Co_2CO_6EP_2$

4.379

2.528

1.756

0.372

1.000

ppm

FIG 2

FIG 3